(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau





(43) International Publication Date 20 March 2003 (20.03.2003)

PCT

(10) International Publication Number WO 03/022983 A1

(51) International Patent Classification7: C12G 1/00, B01F 3/04

C12H 1/22,

(21) International Application Number: PCT/AU02/01250

(22) International Filing Date:

12 September 2002 (12.09.2002)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

PR 7674 PR 8303 13 September 2001 (13.09.2001) 16 October 2001 (16.10.2001)

(71) Applicant (for all designated States except US): OZMOX PTY. LTD. [AU/AU]; 685 Centre Road, Bentleigh, Victo-

(72) Inventors; and

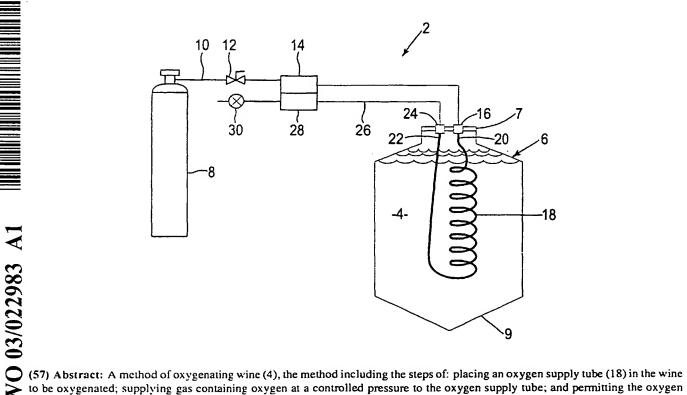
ria 3204 (AU).

(75) Inventors/Applicants (for US only): KELLY, Mark, Aidan [AU/AU]; 30 Hallam Street, Bendigo, Victoria 3550 (AU). WOLLAN, David [AU/AU]; 14 Maverick Close, Doncaster, Victoria 3108 (AU).

- (74) Agents: PRYOR, Geoffrey, Charles et al.; Davies Collison Cave, 1 Little Collins Street, Melbourne, Victoria 3000 (AU).
- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

[Continued on next page]

(54) Title: METHOD AND APPARATUS FOR OXYGENATING WINE



to be oxygenated; supplying gas containing oxygen at a controlled pressure to the oxygen supply tube; and permitting the oxygen from the gas to permeate through the oxygen supply tube to dissolve into the wine.



WO 03/022983 A1



Published:

with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

-1-

METHOD AND APPARATUS FOR OXYGENATING WINE

This invention relates to a method and apparatus for oxygenating wine.

It is known that the quality of the wine can be improved by oxygenating or microoxygenating the wine during the maturation process. Some research suggests that this occurs naturally when wine is stored in wooden barrels.

French Publication No. 2709983 discloses a method of micro-oxygenating wine held in tanks. In practice, the technique is quite complex and requires careful control. The technique involves injecting small bubbles of oxygen into the wine so that it will bubble through the wine so that some of the oxygen will be absorbed during the time in which the bubbles pass through the wine.

There are, however, some drawbacks with this technique. First, quite stringent control is necessary in order to produce suitable fine bubbles. Second, the bubbles need to pass through the wine for a predetermined distance so that sufficient oxygen is absorbed into the wine. Typically this distance is of the order of about two metres. This makes the technique unsuitable for use in small or shallow tanks.

20

`,

The object of the present invention is to provide an alternative method and apparatus which at least partially overcome the drawbacks noted above.

According to the present invention there is provided a method of oxygenating wine, 25 the method including the steps of:

placing an oxygen supply tube in the wine to be oxygenated;

supplying gas containing oxygen at a controlled pressure to the oxygen supply tube; and

permitting the oxygen from the gas to permeate through the oxygen supply tube to dissolve into the wine.

- 2 -

Preferably, the gas is substantially pure oxygen but it could be air.

In the method of the invention, after the oxygen has permeated through the oxygen supply tube, it is directly dissolved into the wine and does not form bubbles in the fluid.

5

٠, ٔ

Preferably further, the method includes the step of supplying oxygen to an inlet end of the tube and the step of monitoring oxygen passing from an outlet end of the tube.

Preferably further, the monitoring includes the step of monitoring the pressure of the oxygen at the inlet and outlet ends of the tube whereby any leak in the tube can be detected.

Preferably further, the monitoring can include the step of detecting traces of the wine in the oxygen discharged from the outlet of the tube.

15

Preferably further, the pressure of the oxygen at the inlet end of the tube is in the range from 0 to 1000 kPa and preferably about 400 kPa.

Preferably further, the tube is made from a polymeric material.

20

Preferably further, the polymeric material is polytetrafluoroethylene (Teflon) or polydimethylsiloxane (Silastic).

Preferably further, the rate at which oxygen is dissolved into and reacted with the wine is in the range 1 to 100 millilitres per litre of wine per month.

Preferably further, the oxygen is first introduced into the wine after initial fermentation and prior to malolactic fermentation (MLF). MLF normally occurs from two to four weeks after primary fermentation. After MLF has ceased, oxygenation can recommence and continue for a period typically in the range from one to six months.

٠,

Preferably further, the rate of supply of oxygen to the wine is decreased as a function of time in the oxygenation which takes place after MLF.

According to the present invention there is also provided an apparatus for oxygenating wine, the apparatus including:

an oxygen supply tube;

means for mounting the supply tube in said wine, and gas supply means for supplying a gas containing oxygen at controlled pressure to the oxygen supply tube; and

wherein the oxygen supply tube is permeable to oxygen, the arrangement being such that oxygen from the gas can permeate through the oxygen permeable tube to dissolve directly into the wine.

Preferably the means for mounting the supply tube in the wine includes couplings which permit an inlet end and an outlet end of the tube to be connected to a tank which holds the wine. Preferably further, the apparatus includes control apparatus for controlling pressure of said gas at the inlet end of the tube. Preferably further, the control means includes a pressure-monitoring device for monitoring the pressure at the outlet end of the tube.

Preferably further, the tube is formed from polymeric material such as polytetrafluoroethylene. Preferably the wall thickness of the tube is in the range from 0.3 to 0.5mm and preferably about 0.3mm.

The invention will now be further described with reference to the accompanying drawings, in which:

Figure 1 is a schematic view of an oxygenating system embodying the principles of the invention;

Figure 2 is a schematic view of a modified arrangement;

Figure 3 is a schematic view of a barrel fitted with a bung device of the invention;

Figure 4 is a side view of the bung device;

Figure 5 is a schematic view of a preferred form of oxygenating system of the

- 4 -

invention;

10

15

20

25

30

÷

Figure 6 is a schematic sectional view through the oxygenating module used in the system of Figure 5; and

Figure 7 is a schematic cross-section through an oxygen permeable tube surrounded by a protective sheath.

Figure 1 diagrammatically illustrates an oxygenating system 2 for oxygenating wine 4 stored in a storage tank 6 which is typically made from stainless steel. Typically the tank 6 includes a removable cover 7 at the top of the tank. The bottom 9 of the tank is also formed in a shallow conical shape. The system includes an oxygen supply cylinder 8 which is coupled to an oxygen supply line 10. The oxygen supply line 10 includes a pressure regulating valve 12 and monitoring apparatus 14. The monitoring apparatus 14 may monitor a number of parameters including pressure, flow rate and may include sensors for impurities or the like. The end of the line 10 is connected to an inlet coupling 16 which is preferably located in the cover 7 of the tank. The system includes a tube 18 having an inlet end 20 which is connected to the inlet coupling 16. The tube 18 is preferably formed from oxygen permeable material such as polytetrafluoroethylene. The tube 18 preferably has an inside diameter of say 3mm and a wall thickness of 0.3mm and a length of 10m. This is suitable for oxygenation of wine where the capacity of the tank 6 is 4000 litres.

In accordance with the invention, oxygen from the cylinder 8 is supplied to the inlet end 20 of the tube 18 at a controlled pressure so that it will permeate through the wall of the tube and be directly dissolved into the wine in which the tube is immersed, without formation of bubbles. It is possible to have the other end 22 of the tube closed so that all of the oxygen which flows into the tube eventually permeates therethrough. Whilst this arrangement would function, it is preferable to have the other end 22 of the tube connected to an outlet coupling 24 which in turn is connected to an oxygen outlet line 26. The outlet coupling 24 is also located in the cover 7 of the tank. The location of the couplings 16 and 24 in the cover 7 minimises the possibility of wine leakage from the tank 6.

٠,

The line 26 includes a valve 30 to assist in maintaining pressure in system. The outlet line 26 may include monitoring apparatus 28 for monitoring parameters such as pressure and composition of the gas flowing in the outlet line 26. By having the monitoring apparatus 28 provided, it is possible to monitor the differences in pressure in the supply line 10 and the outlet line 26. If there is a large pressure differential this could indicate that the tube 18 has ruptured or blocked which could cause spoiling of the wine by supplying excess or insufficient oxygen thereto. In one convenient arrangement, the monitoring apparatus 14 and 28 can take the form of a single differential pressure unit which senses differences in pressure in the lines 10 and 26. If a large pressure differential is detected, that could be used to change the state of a switch which in turn could be used to close a solenoid valve (not shown) or the like in the inlet line 10 to stop the flow of oxygen in the inlet line 10. This would, of course, substantially reduce the risk of spoiling of the wine by over oxygenation thereof should a rupture occur in the tube 18 or in the couplings and lines to which the tube 18 is connected. Further, the monitoring apparatus may include sensors for sensing traces of wine in the line 26 which again would be indicative of leaks in the tube 18.

The flow rate for permeation of oxygen through the tube 18 is governed by the following formula:

$$flow rate = \mu \cdot \frac{A}{t} \cdot \Delta P \tag{1}$$

where A is the surface area of the tube 18;

t is the wall thickness;

μ is the specific permeability of the tube material; and

 ΔP is the pressure differential across the tube wall.

25

30

15

In the system of the invention, the pressure regulating valve 12 can be adjusted so as to provide the required pressure differential for a desired flow rate of oxygen into the wine. Typically the pressure of the oxygen in the supply line 10 would be in the range from 0 to 1000 kPa and more generally about 400 kPa, although this would also depend on the tube surface area and wall thickness.

- 6 -

For maturation of wine, the periods during which oxygenation takes place and the rates of oxygen flow into the wine are varied by the wine maker in accordance with individual preferences and the styles of wines which are to be produced. Experience with the micro-oxygenation technique disclosed in the aforementioned French publication suggests that it is preferable to oxygenate the wine after primary fermentation has ceased but before the onset of MLF. After MLF ceases, a second period of oxygenation may occur at a rate which is comparable with the rate in the initial period but may be reduced as maturation continues. Typically the second period of treatment lasts from one to six months.

10

:

Because the wall thickness of the tube 18 is relatively thin, the tube is somewhat fragile and susceptible to damage. Accordingly, it may be contained within a protective bag, sheath or container (not shown) which serves to protect the tube 18 from physical damage but does not substantially impede flow of wine and/or oxygen therethrough.

15

20

25

In the preferred method of the invention, pure oxygen is supplied from the oxygen supply cylinder 8. It would, however, be possible to use other gases which contain oxygen. For instance, air could be supplied to the tube 18 but this would be less efficient because other components of the air, chiefly nitrogen gas, would also permeate through the tube 18. Thus a longer tube or higher supply pressure would be needed. Accordingly, it is preferred to use pure or substantially pure oxygen for supply to the tube 18.

Figure 2 schematically illustrates a modified form of the invention. In this arrangement a composite tube 32 is provided which can be operated so that it can vary the effective length thereof. This is accomplished by having a second oxygen inlet line 34 coupled to the inlet end 36 of a second oxygen permeable tube 38, which is preferably made from the same material as the tube 18 but of a different length, say two to five times as long as the tube 18. The outlet end 40 of the tube 38 is connected via a T-piece 42 to the inlet end 20 of the tube 18 via a check valve 41. In a first mode, oxygen is supplied via the oxygen supply line 10 (and not through the second inlet line 34) so that oxygen flows through the tube 18 as before. The check valve 41 prevents flow of oxygen into the second

- 7 -

tube 38. If it is determined that additional oxygen is required, the composite tube 32 can be used in a second mode in which oxygen is supplied to the second oxygen inlet line 34 and not to the supply line 10 which would be closed. In this case the oxygen flows through the second tube 38 through the check valve 41 and T-piece 42 and then into the tube 18. The effective length of the tube is much greater and hence the rate of oxygen absorption will be greater in the wine in which the composite tube 32 is immersed.

Figure 3 shows an oxygenating bung device 43 of the invention which is designed for use with a wine barrel 44. Generally speaking, the bung device 43 can be used where the wine is being stored in barrels rather than a large tank as in the case of Figure 1. The bung device 43 includes a bung 45 which is initially of standard size and shape. Typically the bung 45 is made of silicone rubber and may have a narrow end of say 48mm and an upper end of 60mm, with a depth of about 55mm. Two bores 46 and 47 are moulded in the bung 45. The bores 46 and 47 snugly receive supply and return tubes 48 and 49 which are made from material which is impermeable to oxygen. Typically the tubes are made from nylon, polypropylene or other oxygen impermeable material. The outer ends of the tubes 48 and 49 are connected to the oxygen supply line 10 and oxygen outlet line 26 respectively. The system is otherwise similar to that shown in Figure 1. The oxygen permeable tube 18 is connected to the inner ends of the tubes 48 and 49, as shown in 20 Figure 4. Couplings could be used to connect the ends of the tube 18 to the tubes 48 and 49. In the illustrated arrangement, however, the ends of the tube 18 are resiliently expanded so as to slide over and sealingly engage the ends of the tubes 48 and 49. This forms a suitable coupling. The length of the tube 18 can be selected in accordance with the size of the barrel 44 and desired rate of oxygenation.

25

30

5

In the bung device 43, the tube 18 preferably has a diameter to wall thickness ratio of approximately 10:1. The tube 18 may be 8mm external diameter. The tubes 48 and 49 preferably have the same external diameter as the tube 18. The length of the tubes 48 and 49 is typically about 250mm each. As mentioned previously, the length of the tube 18 can be adjusted in accordance with the volume of the barrel 44. For a barrel of 225 litres capacity an oxygenation rate of say 10mls per litre per month can be achieved where the

- 8 -

length of the tube 18 is 250mm when the supply tube 48 is supplied with air at atmospheric pressure. An air pump or fan (not shown) can be coupled to the tube 48 or 49 in order to circulate air through the tube 18. The rate of oxygenation can be increased by increasing the oxygen percentage in the feed gas supplied to the tube 48. The rate of oxygenation can also be reduced by supplying less oxygen rich gases to the tube 48. This can be achieved by operating a feed pump (not shown) so that the supply of oxygen containing gas is varied as a function of time. The combination of supply rate of oxygen containing gas and consumption rate of oxygen reduces the average oxygen concentration in the permeable tube.

10

15

20

25

`

Figure 5 illustrates another oxygenating system of the invention. In this embodiment, the same reference numerals have been used to denote parts which are the same as or correspond to those of the previous embodiments.

In the arrangement illustrated in Figure 5, the system includes an oxygen supply module 50 which is suspended in the storage tank 6 so that it is completely immersed in the wine 4. Preferably, the module 50 is located near the bottom of the tank. The module 50 will normally be located about 200mm from the bottom of the tank so that it is clear of the lees which may accumulate in the bottom of the tank. Normally the module 50 would not be more than say one metre above the bottom of the tank. In the illustrated arrangement, the module 50 includes a loop 52 which enables it to be suspended by a line 54 which in turn is connected to the underside of the cover 7 of the tank 6. The module 50 includes inlet and outlet connectors 56 and 58 which enable the module to be coupled to the inlet and outlet couplings 16 and 24 by means of inlet supply tubing 60 and outlet tubing 62. The module 50 includes means for circulating the wine therethrough so that it flows over an oxygen permeable tube located inside the module. This enables the oxygen to be directly absorbed into the wine without forming bubbles.

As mentioned above, the module includes a pump or impeller for circulating the wine through the module. It is possible to mount on or in the module 50, a container 63 in which can be located additives. The additives may, for instance, comprise oak chips or

:

20

- 9 -

other additives. In the illustrated arrangement the container 63 can be formed from stainless steel mesh or other material which is inert to wine. It is disposed so that wine flows through the container 63 as wine flows to or from the module. This provides a very convenient way in which the wine can be treated with oak chips or other additives whilst the oxygenation is also taking place.

Figure 6 shows in part section in more detail a module 51 which can be used in the system shown in Figure 5. The module 51 may also have a container (not shown in Figure 6) associated with it similar to the container 63 described above for holding oak chips or additives. The module 51 includes a coil housing 70 which is formed by upper and lower disc members 72 and 74. A perforated stainless steel mesh 76 extends between the disc members 72 and 74. The oxygen tube 18 is formed into a helically wound coil 78 located within the housing 70. The tube 18 functions in the same way as that of the previous embodiments and therefore need not be described in detail. The convolutions of the coil 78 are held spaced from one another by means of a spacing grid (not shown) or radiantly extending baffles (not shown). This enables good wine circulation through the coil 78. The lower disc member 74 includes a central inlet opening 80 which opens to an impeller chamber 82 in which is mounted an impeller 84. As will be described in more detail below, the impeller 84 is rotated so as to draw wine through the opening 80 into the chamber 82 and then radially outwardly passing over the convolutions of the coil 78, the wine being expelled through an outlet defined by the perforated mesh 76. This ensures that there is good circulation of wine through the module 51 so that all of the wine in the tank 6 can be uniformly treated.

The impeller 84 is mounted on an impeller shaft 86, the lower end of which is supported by a lower bearing 88. The lower bearing 88 can be integrally formed with disc member 74. This would be suitable in the case where the disc member 74 (as well as the disc member 72) were made from nylon. It would be possible, also, to provide a separate teflon insert (not shown) to serve as a bearing for the shaft 86. The upper end of the shaft 86 is supported by means of a bearing 90. The upper face of the impeller 84 is provided with a plurality of magnets 92 to enable magnetic coupling to a drive motor 94. The motor

5

15

₹.

94 is located in a motor housing 96 which, generally speaking, is mounted centrally of the upper disc member 72.

The motor housing 96 includes a main block 98 having a cylindrical recess 100 therein in which the motor 94 is mounted. The motor has an output shaft 107 upon which a drive disc 106 is mounted. The lower face of the block 98 includes a cylindrical mounting portion 102 which is received within a complementary opening in the centre of the upper disc member 72. A skirt portion 104 extends downwardly from the mounting portion 102. The drive disc 106 has mounted thereon a plurality of magnets 108 and is located in the skirt portion 104. The skirt portion 104 has a relatively thin bottom wall 108 which forms a fluid tight barrier at the bottom of the recess 100. The thickness of the wall 108 is preferably less than 2mm. This enables the magnets 92 of the impeller to be closely spaced relative to the magnets 105 of the disc and hence the motor 94 is magnetically coupled to drive the impeller 84. The bearing 90 can be integrally formed at the centre of the wall 108. The housing 96 includes a cover 110 which is formed with a central tubular spigot 112. The spigot 112 serves as an entrance point for the oxygen supply and return lines to the coil 78 as well as electric conductors for the motor 94. In the illustrated arrangement, the module 51 includes an oxygen supply line 114 which extends through bores provided in the cover 110 and main block 98 so as to be coupled to the inlet end 116 of the coil 78. The module 51 includes an oxygen return line 118 which again passes through bores in the cover 110 and block 98 so as to be connected to the outlet end 120 of the coil 78.

A sheath 126 surrounds the lines 114 and 118 and in use is connected to the spigot
112 by means of a clamp (not shown). This serves to protect the lines and conductors and
may also serve as the means by which the module 51 can be suspended in the tank. In the
illustrated arrangement, four mounting bolts 128 extend through the housings 70 and 96 in
order to hold its components together. Briefly, the bolts 128 extend from the lower disc
member 74 and then through the upper disc member 72 and then through the block 98 and
cover 110.

In the illustrated arrangement, the impeller 84 is rotated at a speed of say 1500rpm. It has been found that the impeller 84 can be a smooth disc like member which has a pumping effect by means of frictional contact with the wine. The wine will be drawn into the chamber 82 through the opening 80 and then radially outwardly passing between the convolutions of the coil 78 to be discharged through the perforated mesh 76. As mentioned above, the motor 94 is mounted in the recess 100 which is sealed so that wine cannot enter the recess. The motor 94 is preferably a DC motor operating at say 24 volts and rated at 7 watts. The size of the motor can, of course, be varied in accordance with requirements. Typically, the disc members 72 and 74 are both made from nylon as is the motor housing 96. The diameter of the disc members 72 and 74 can be say 150mm and the overall height of the module about 100mm. The diameter of the impeller 84 may typically be 30mm. Where a larger module is made, it may be desirable to put a surface pattern on the impeller 84 or to form it with blades or the like to enhance the pumping action. The length of the tube 18 which forms the coil 78 can be varied in accordance with requirements. Typically it is 3mm external diameter and having a length in the range 1m to 20m.

10

15

20

The module 51 shown in Figure 6 has a significant advantage over the arrangement shown in Figure 1 in that the coil 78 is protected within the module and hence it is much less susceptible to damage. Also, the provision of the pump ensures good circulation of wine so that dead spots can be eliminated and unwanted build-up of oxygen bubbles on the surface of the tube is avoided.

The flow rate of wine through the module can be varied according to requirements.

It is envisaged that quite a low flow rate would be required because of the amount of time in which the module would be used in the tank. A flow rate of about 40 litres per minute has been tested in a prototype and found to be satisfactory.

A new highly oxygen permeable polymeric material has recently been developed and is known as amorphous polytetrafluoroethylene. One type of this material is known as Teflon AF made by DuPont and it has an oxygen permeability many times greater than that

- 12 -

of other forms of polytetrafluoroethylene.

ì

10

Theoretically, Teflon AF would be very suitable for use as the tube 18 in the various embodiments of the invention. At the present time, however, there are a number of drawbacks associated with Teflon AF. First, it is very expensive. Second, it is very fragile. Third, it is not readily available. Fourth, it currently does not have US FDA approval for food contact use. Notwithstanding these drawbacks, preliminary studies show that it would offer significant advantages in the system of the invention. For instance, it has been calculated that the tube 18 could be formed of Teflon AF with a 1.5mm internal diameter and 0.15 wall thickness. A 300mm length of this tubing supplied with oxygen at STP would provide an oxygenation rate of 10mm per litre of wine per month for a tank of capacity of 25,000 litres. In the module 51 shown in Figure 6, a single coil of this material would be all that is required within the housing 70.

In accordance with the invention, it is thought that a number of the disadvantages potentially associated with Teflon AF can be overcome by the arrangement diagrammatically shown in Figure 7.

Figure 7 illustrates an alternative arrangement for the tube 18 of the invention. In this arrangement the tube 18 is made from Teflon AF, say having an internal diameter of 1.5mm and a wall thickness of 0.15mm. This material is thought to be strong enough to handle the internal pressure of the oxygen supplied to its inner bore. It is, however, somewhat prone to kinking which leads to fracture. Accordingly, it is inserted within a sheath 90 of oxygen permeable material which supports the tube 18 and makes it less susceptible to kinking. The sheath 90 has an internal diameter of say 2mm and a wall thickness of say 0.05mm. A gap 92 is therefore defined between the outer diameter of the tube 18 and the internal diameter of the sheath 92. In use this gap would be filled with oxygen which permeates through the tube 18. The sheath 90 is such that it offers little resistance to the permeation of oxygen therethrough so that the oxygen will dissolve into the wine which surrounds the sheath 90.

٠.

15

20

- 13 -

The preferred material for the sheath 90 is expanded polytetrafluoroethylene which is known as ePTFE or porous PTFE. When the arrangement of Figure 7 is utilised in accordance with the invention, the rate of supply of oxygen to the tube 18 is such that the oxygen which passes through the sheath 90 is directly absorbed into the wine and tends not to form any bubbles. PTFE is also very suitable for this purpose because it is hydrophobic and therefore also tends to resist formation of bubbles of oxygen. The ePTFE material which forms the sheath 90 preferably has an average internode gap of 23 (IND 23). Although oxygen can permeate through the sheath 90 with little impediment, water cannot permeate the material of the sheath and hence the wine tends not to enter into the gap 92. Accordingly, the tube 18 is not normally directly in contact with the wine because of the existence of the sheath 90.

The reference to any prior art in this specification is not, and should not be taken as, an acknowledgment or any form of suggestion that that prior art forms part of the common general knowledge.

The principles of the invention are applicable to oxygenation of products other than wine. For instance, the techniques of the invention could be used in various fermentation processes or other chemical processing reactions where small controlled quantities of oxygen need to be released into a fluid.

Many modifications will be apparent to those skilled in the art without departing from the spirit and scope of the invention.

;

CLAIMS:

- 1. A method of oxygenating wine (4), the method including the steps of: placing an oxygen supply tube (18) in the wine to be oxygenated;
- supplying gas containing oxygen at a controlled pressure to the oxygen supply tube; and

permitting the oxygen from the gas to permeate through the oxygen supply tube to dissolve into the wine.

- 10 2. A method as claimed in claim 1 wherein after the oxygen has permeated through the oxygen supply tube, it is dissolved into the wine and does not form bubbles in the wine.
 - 3. A method as claimed in claim 1 or 2 wherein the oxygen supply tube is immersed in the wine.

- 4. A method as claimed in claim 1, 2 or 3 wherein the gas is oxygen.
- 5. A method as claimed in claim 1, 2 or 3 wherein the gas is air.
- 6. A method as claimed in any one of claims 1 to 5 wherein the rate at which oxygen is dissolved into the wine is varied by selecting the surface area of the tube which is in contact with the wine and selecting the pressure of said gas.
- 7. A method as claimed in any one of claims 1 to 6 wherein the wine is stored in a tank having a bottom and the method includes the step of locating the supply tube near the bottom of the tank.
 - 8. A method as claimed in claim 7 wherein the rate of dissolution of oxygen into the wine is in the range of 1 to 100 millilitres at STP per litre of wine per month.

- 9. A method as claimed in claim 7 or 8 including the steps of commencing oxygenation after initial fermentation and stopping oxygenation before malolactic fermentation of the wine takes place.
- 5 10. A method as claimed in claim 9 including the step of recommencing oxygenation after malolactic fermentation of the wine has ceased.
 - 11. A method as claimed in claim 10 including the step of reducing the rate of oxygenation as a function of time after recommencing oxygenation.
 - 12. A method as claimed in any one of claims 1 to 11 wherein the oxygen supply tube (18) is located within a protective sheath (90).
- 13. A method as claimed in claim 12 wherein the protective sheath is permeable to oxygen but impermeable to wine.
 - 14. A method as claimed in any one of claims 1 to 13 including the steps of supplying the gas containing oxygen through an oxygen supply line (10) to an inlet end (20) of the oxygen supply tube and connecting an oxygen outlet line (26) to the other end (22) of the tube (18).
 - 15. A method as claimed in claim 14 including the step of monitoring the pressure of the gas in said supply and the outlet lines.
- 25 16. A method as claimed in claim 14 including the step of monitoring the differential pressure of said gas in said supply and outlet lines.
 - 17. A method as claimed in claim 16 including the step of stopping the supply of gas in said supply line if a differential pressure above a predetermined magnitude is detected.

10

20

:

٠,

20

18. Apparatus for oxygenating wine, the apparatus including: an oxygen supply tube (18);

means (16,24,45,50,51) for mounting the supply tube in said wine, and gas supply means (8,12,14) for supplying a gas containing oxygen at controlled pressure to the oxygen supply tube; and

wherein the oxygen supply tube is permeable to oxygen, the arrangement being such that oxygen from the gas can permeate through the oxygen permeable tube to dissolve directly into the wine.

- 19. Apparatus as claimed in claim 18 wherein the means for mounting the supply body in the wine includes couplings (16,24) which permit an inlet end (20) and an outlet end (22) of the tube to be connected to a tank (6) which holds the wine.
- 20. Apparatus as claimed in claim 18 or 19 wherein the means for mounting the supply body in the wine includes couplings (16,24) which permit an inlet end (20) and an outlet end (22) of the tube to be connected to a tank (6) which holds the wine.
 - 21. Apparatus as claimed in claim 18, 19 or 20 wherein the apparatus includes control apparatus (12) for controlling pressure of said gas at the inlet end of the tube.
 - 22. Apparatus as claimed in any one of claims 18 to 21 wherein the tube is formed from polymeric material.
- 23. Apparatus as claimed in claim 22 wherein the polymeric material is polydimethylsiloxane or polytetrafluoroethylene.
 - 24. Apparatus as claimed in any one of claims 18 to 23 wherein the ratio of diameter to wall thickness is about 10:1.
- 30 25. Apparatus as claimed in any one of claims 18 to 24 wherein the wall thickness of the tube is in the range 0.5mm to 0.375mm.

- 26. Apparatus as claimed in any one of claims 18 to 25 wherein the gas supply means includes an oxygen supply cylinder (8) for supplying substantially pure oxygen to the tube.
- 27. Apparatus as claimed in claim 26 wherein the gas supply means supplies oxygen at a pressure in the range from 0 to 1000 kPa to an inlet end of the tube.
 - 28. Apparatus as claimed in any one of claims 18 to 27 wherein the gas supply means includes a supply line (10) for supplying said gas to an inlet end (20) of the tube, said apparatus also including a gas return line (26) coupled to an outlet end (22) of the tube.
 - 29. Apparatus as claimed in claim 28 including monitoring means (14,28) for monitoring the pressure differential of gas in the supply and return lines.

10

- 30. Apparatus as claimed in claim 29 including control means coupled to the monitoring means to stop flow of gas in the supply line if a differential pressure above a predetermined magnitude is detected.
 - 31. Apparatus as claimed in any one of claims 18 to 30 wherein the oxygen supply tube (18) is located within a protective sheath (90).
 - 32. Apparatus as claimed in claim 31 wherein the protective sheath is permeable to oxygen but impermeable to wine.
- 33. Apparatus as claimed in claim 31 or 32 wherein the oxygen supply tube comprises Teflon AF.
 - 34. Apparatus as claimed in claim 33 wherein the protective sheath comprises ePTFE.
- 35. Apparatus for oxygenating wine stored in a barrel (44) having a bung hole therein, 30 the apparatus including:
 - a bung (45) which is adapted to fit in the bung hole;

an inlet tube (48) passing longitudinally through the bung;

an outlet tube (49) passing longitudinally through the bung, said inlet and outlet tubes being made from oxygen impermeable material;

an oxygen supply tube (18) made from oxygen permeable material having ends connected to respective ends of the inlet and outlet tubes, the arrangement being such that in use the oxygen supply tube is immersed in the wine in the barrel and whereby oxygen from an oxygen containing gas supplied to said inlet tube permeates through the oxygen supply tube to dissolve directly into the wine.

10 36. A module (50,51) for immersion in wine in a wine storage tank, said module including:

a coil housing (70) for containing a coil (78) of oxygen permeable tube (18);

said housing including an inlet (80) and outlet (76) for defining a flow path for wine through the housing; and

pumping means (84) for causing wine to flow through the housing along said flow path, and

gas supply means (114) for coupling a supply of a gas containing oxygen to an inlet end of said coil, the arrangement being such that oxygen in said gas permeates through the tube to be dissolved into wine flowing through the housing.

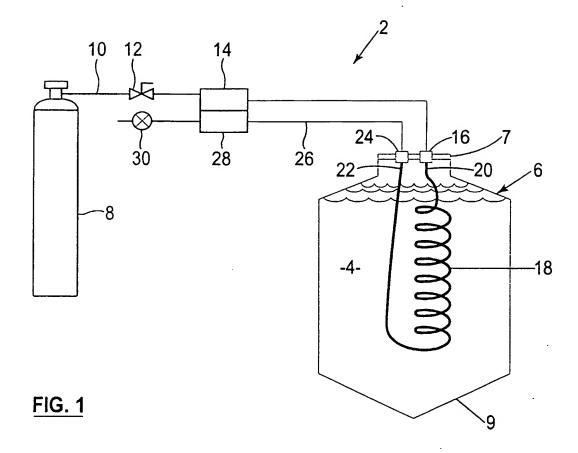
20

- 37. A module as claimed in claim 36 wherein the pumping means includes a motor (94) coupled to an impeller (84) located in an impeller chamber (82) within the housing.
- 38. A module as claimed in claim 37 wherein the impeller is magnetically coupled to an output shaft (107) of the motor (94).
 - 39. A module as claimed in claim 38 wherein the module includes a motor (94) located in a sealed chamber (100) located in a motor housing (96).

- 40. A module as claimed in claim 39 wherein a disc (106) carrying first magnets (105) is mounted on the output shaft (107) and second magnets (92) are carried by the impeller (84).
- 5 41. A module as claimed in any one of claims 36 to 40 wherein the coil housing (70) includes upper and lower discs (72,74) having a perforated mesh (76) therebetween which defines said outlet and wherein the lower disc (74) includes a central opening (80) which defines said inlet.
- 10 42. A module as claimed in claim 39 wherein the motor housing (96) is mounted on the coil housing (70), the motor housing including a hollow spigot (112).
 - 43. A module as claimed in claim 42 wherein the gas supply means includes an inlet line (114) for coupling to an inlet end (116) of the coil and a gas return line (118) which is connected to an outlet end (120) of the coil and wherein said inlet and return lines pass through said hollow spigot.
- 44. A module as claimed in claim 43 including electric conductors (122,124) for supplying operating current to the motor and wherein the conductors pass through said
 20 hollow spigot.
 - 45. A module as claimed in claim 44 including a sheath (126), a lower end of which is coupled to the spigot and wherein the inlet and outlet lines and said conductors are located within the sheath.
 - 46. A module as claimed in any one of claims 41 to 45 wherein the oxygen supply tube (18) is located within a protective sheath (90).
- 47. A module as claimed in claim 46 wherein the protective sheath is permeable to 30 oxygen but impermeable to wine.

- 20 -

- 48. A module as claimed in claim 46 or 47 wherein the oxygen supply tube comprises Teflon AF.
- 49. A module as claimed in claim 48 wherein the protective sheath comprises ePTFE.



2/7

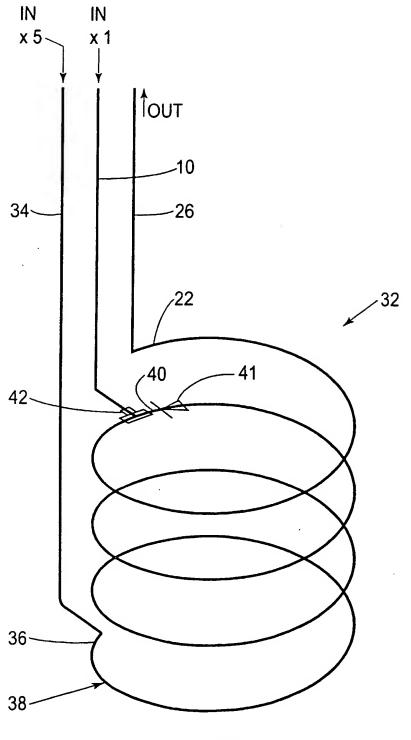
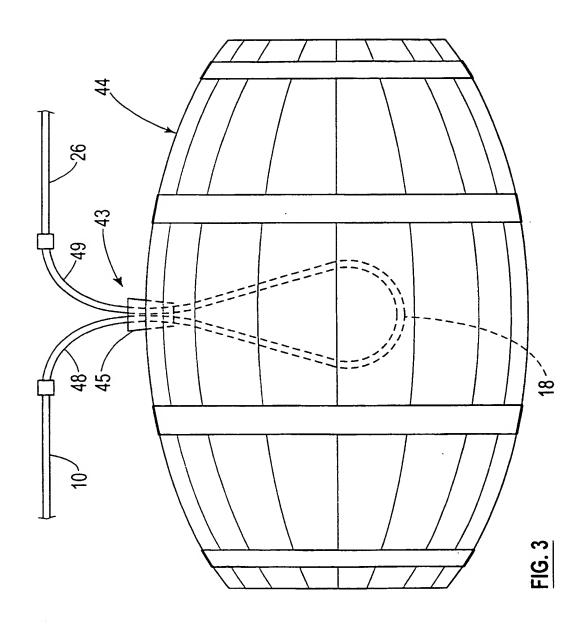


FIG. 2

SUBSTITUTE SHEET (RULE 26)



4/7

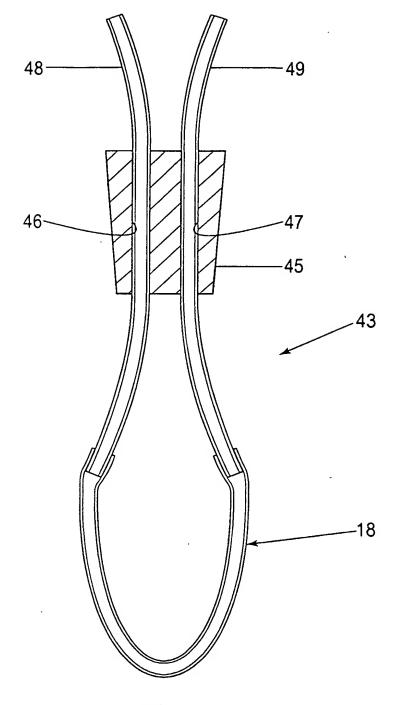
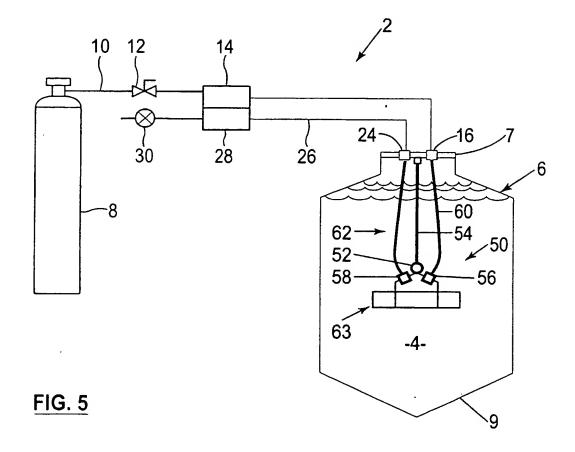


FIG. 4



6/7

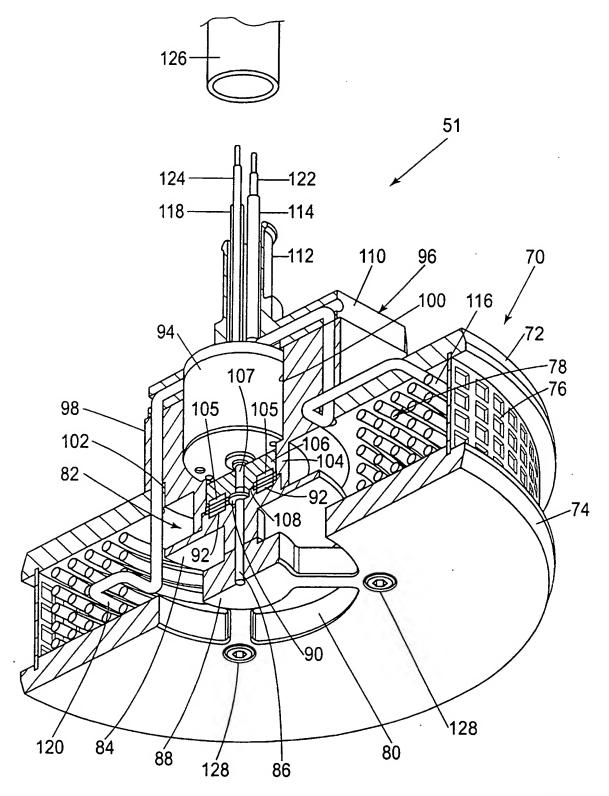


FIG. 6

7/7

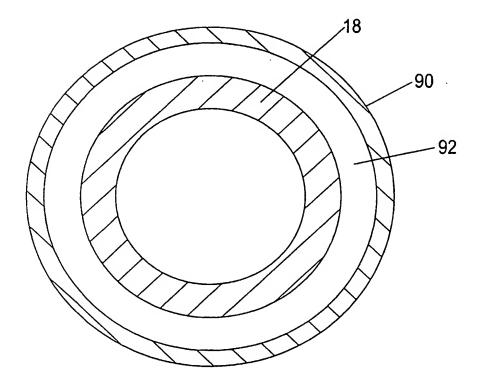


FIG. 7

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU02/01250

CLASSIFICATION OF SUBJECT MATTER Int. Cl. 7: C12H 1/22, C12G 1/00, B01F 3/04 According to International Patent Classification (IPC) or to both national classification and IPC FIELDS SEARCHED B. Minimum documentation searched (classification system followed by classification symbols) C12G 1/-, C12H 1/-, B01F, B01J 4/-Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) WPIDS: wine, alcohol, fermentation, oxygen, air, aerate, permeate, diffuse DOCUMENTS CONSIDERED TO BE RELEVANT C. Relevant to Citation of document, with indication, where appropriate, of the relevant passages Category* claim No. EP 1 083 221 A (Forschungszentrum Jülich GmbH) 14 March 2001 1-34 Х whole of document JP 02-249478 A(Morita KK) 5 October 1990 1-7, 12-34 Х abstract US 6 378 419 B (Ecklein) 30 April 2002 1-5, 7-11, 14, P, X whole of document 15, 18-21, 26-28 See patent family annex $|\mathbf{x}|$ Further documents are listed in the continuation of Box C Special categories of cited documents: later document published after the international filing date or priority date **"**A" document defining the general state of the art and not in conflict with the application but cited to understand the principle which is not considered to be of particular or theory underlying the invention document of particular relevance; the claimed invention cannot be "E" earlier application or patent but published on or considered novel or cannot be considered to involve an inventive step after the international filing date when the document is taken alone document of particular relevance; the claimed invention cannot be "L" document which may throw doubts on priority considered to involve an inventive step when the document is combined claim(s) or which is cited to establish the with one or more other such documents, such combination being obvious to publication date of another citation or other special reason (as specified) a person skilled in the art document member of the same patent family "O" document referring to an oral disclosure, use, exhibition or other means document published prior to the international filing date but later than the priority date claimed Date of mailing of the international search report Date of the actual completion of the international search 1 8 OCT 2002 14 October 2002 Authorized officer Name and mailing address of the ISA/AU **AUSTRALIAN PATENT OFFICE** PO BOX 200, WODEN ACT 2606, AUSTRALIA **GARETH COOK** E-mail address: pct@ipaustralia.gov.au Telephone No: (02) 6283 2541 Facsimile No. (02) 6285 3929

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU02/01250

	tion). DOCUMENTS CONSIDERED TO BE RELEVANT	Relevant to					
ategory*	Citation of document, with indication, where appropriate, of the relevant passages						
	WO 95/00401 A (Intellection) 5 January 1995						
Α	whole of document						
	EP 526 823 A (Japan Gore-Tex) 10 February 1993						
Α	whole of document						
	US 3 942 423 A (Herzfeld) 9 March 1976						
Α	whole of document						
•							
•							

Information on patent family members

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
EP	1 083 221	DE	19 938 226				
wo	95/00401	AU	62261/94	NZ	2 631 129	US	5 537 913
EP	526 823	DE	69 202 022	JР	5 038 424	us	5 262 096
			·				END OF ANNEX